

EARLY ENTRANCE COPRODUCTION PLANT

PHASE II

Topical Report

Task 2.10: Environmental

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Abstract

The overall objective of this project is the three phase development of an Early Entrance Coproduction Plant (EECP) which uses petroleum coke to produce at least one product from at least two of the following three categories: (1) electric power (or heat), (2) fuels, and (3) chemicals using ChevronTexaco's proprietary gasification technology. The objective of Phase I is to determine the feasibility and define the concept for the EECP located at a specific site; develop a Research, Development, and Testing (RD&T) Plan to mitigate technical risks and barriers; and prepare a Preliminary Project Financing Plan. The objective of Phase II is to implement the work as outlined in the Phase I RD&T Plan to enhance the development and commercial acceptance of coproduction technology. The objective of Phase III is to develop an engineering design package and a financing and testing plan for an EECP located at a specific site.

The project's intended result is to provide the necessary technical, economic, and environmental information needed by industry to move the EECP forward to detailed design, construction, and operation. The partners in this project are Texaco Energy Systems LLC or TES (a subsidiary of ChevronTexaco), General Electric (GE), Praxair, and Kellogg Brown & Root (KBR) in addition to the U.S. Department of Energy (DOE). TES is providing gasification technology and Fischer-Tropsch (F-T) technology developed by Rentech, GE is providing combustion turbine technology, Praxair is providing air separation technology, and KBR is providing engineering.

During Phase I the team identified several potential methods to reduce or minimize the environmental impact of the proposed EECP. The EECP Project Team identified F-T catalyst disposal, beneficial gasifier slag usage (other than landfill), and carbon dioxide recovery for the gas turbine exhaust for study under this task. Successfully completing the Task 2.10 RD&T provides additional opportunities for the EECP to meet the goals of DOE's Vision 21 Program.

The gasification section offers several opportunities to maximize the environmental benefits of an EECP. The spent F-T catalyst can be sent to landfills or to the gasification section. Testing in Phase II shows that the spent F-T catalyst with a small wax coating can safely meet federal landfill requirements. As an alternative to landfilling, it has been proposed to mix the spent F-T catalyst with the petroleum coke and feed this mixture to the gasification unit. Based on ChevronTexaco's experience with gasification and the characteristics of the spent F-T catalyst this appears to be an excellent opportunity to reduce one potential waste stream. The slag from the gasification unit can be commercially marketed for construction or fuel (such as cement kiln fuel) uses. The technical and economic benefits of these options must be reviewed for the final EECP before incorporating a specific alternative into the design basis.

Reducing greenhouse gas emissions, particularly carbon dioxide, is an important goal of the EECP. The Texaco gasification process provides opportunities to capture high purity streams of carbon dioxide. For Phase II, a carbon fiber composite molecular sieve (CFCMS) was tested to determine its potential to remove high purity carbon dioxide from the exhaust of a gas turbine. Testing on with a simulated gas turbine exhaust shows that the CFCMS is able to remove high purity carbon dioxide from the exhaust. However, more development is required to optimize the system.

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Executive Summary

The overall objective of this project is the three phase development of an Early Entrance Coproduction Plant (EECP) which uses petroleum coke to produce at least one product from at least two of the following three categories: (1) electric power (or heat), (2) fuels, and (3) chemicals using ChevronTexaco's proprietary gasification technology. The objective of Phase I was to determine the feasibility and define the concept for the EECP located at a specific site; develop a Research, Development, and Testing (RD&T) Plan for implementation in Phase II; and prepare a Preliminary Project Financing Plan. The objective of Phase II is to implement the work as outlined in the Phase I RD&T Plan to enhance the development and commercial acceptance of coproduction technology. The objective of Phase III is to develop an engineering design package and a financing and testing plan for an EECP located at a specific site. The project's intended result is to provide the necessary technical, economic, and environmental information needed by industry to move the EECP forward to detailed design, construction, and operation.

During Phase I the team identified several potential methods to reduce or minimize the environmental impact of the proposed EECP. The EECP Project Team identified Fischer-Tropsch (F-T) catalyst disposal, beneficial gasifier slag usage (other than landfill), and carbon dioxide recovery for the gas turbine exhaust for study under this task. Successfully completing the Task 2.10 RD&T provides additional opportunities for the EECP to meet the goals of DOE's Vision 21 Program.

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Background

The overall objective of this project is the three phase development of an Early Entrance Coproduction Plant (EECP) which uses petroleum coke to produce at least one product from at least two of the following three categories: (1) electric power (or heat), (2) fuels, and (3) chemicals. The objective of Phase I was to determine the feasibility and define the concept for the EECP located at a specific site; develop a Research, Development, and Testing (RD&T) Plan for implementation in Phase II; and prepare a Preliminary Project Financing Plan. The objective of Phase II is to implement the work as outlined in the Phase I RD&T Plan to enhance the development and commercial acceptance of coproduction technology. The objective of Phase III is to develop an engineering design package and a financing and testing plan for an EECP located at a specific site. The project's intended result is to provide the necessary technical, economic, and environmental information needed by industry to move the EECP forward to detailed design, construction, and operation.

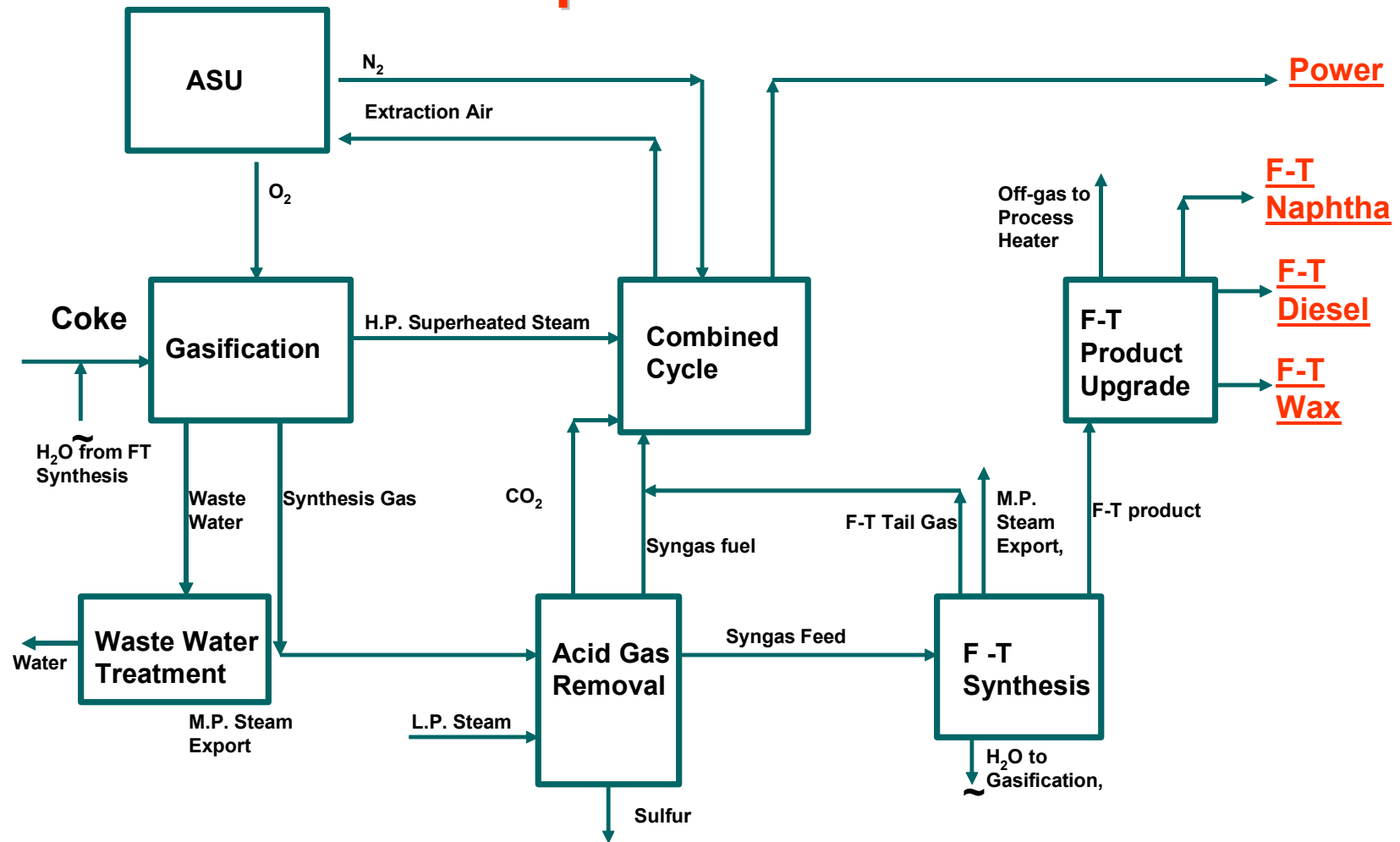
The proposed EECP facility will coproduce electric power and steam for export and internal consumption, finished high-melt wax, finished low-melt wax, Fischer-Tropsch (F-T) diesel, F-T naphtha, elemental sulfur, and consume approximately 1,120 metric tons per day (1,235 short tons per day) of petroleum coke. During Phase I, the Motiva Port Arthur Refinery site was chosen for the EECP. The refinery site offered a ready source of petroleum coke as a feedstock. However, as a result of the merger between Texaco and Chevron, this site is no longer available.

EECP Concept

As shown in Schematic 1, petroleum coke is ground, mixed with water and pumped as thick slurry to the Gasification Unit. This petroleum coke slurry is mixed with high-pressure oxygen from the Air Separation Unit (ASU) and a small quantity of high-pressure steam in a specially designed feed injector mounted on the gasifier. The resulting reactions take place very rapidly to produce synthesis gas, also known as syngas, which is composed primarily of hydrogen (H_2), carbon monoxide (CO), water vapor (H_2O), and carbon dioxide (CO_2) with small amounts of hydrogen sulfide (H_2S), methane, argon (Ar), nitrogen (N_2), and carbonyl sulfide. The raw syngas is scrubbed with water to remove solids, cooled, and then forwarded to the Acid Gas Removal Unit (AGR), where the stream is split. One portion of the stream is treated in the AGR to remove CO_2 and H_2S and then forwarded to the F-T Synthesis Unit. The other portion is treated in the AGR to remove the bulk of H_2S with minimal CO_2 removal and then forwarded as fuel to the GE frame 6FA gas turbine. In the AGR solvent regeneration step, high pressure nitrogen from the ASU is used as a stripping agent to release CO_2 . The resulting CO_2 and nitrogen mixture and the bulk of the nitrogen are also sent to the gas turbine, which results in increased power production and reduced nitrogen oxides (NOx) emissions.

Overall, approximately 75% of the sweetened syngas is sent to the gas turbine as fuel. The remaining 25% is first passed through a zinc oxide bed arrangement to remove the remaining traces of sulfur and then forwarded to the F-T Synthesis Unit. In the F-T reactor, CO and H_2

Proposed EECF



Schematic 1 – EECF Concept

react, aided by an iron-based catalyst, to form mainly heavy straight-chain hydrocarbons. Since the reactions are highly exothermic, cooling coils are placed inside the reactor to remove the heat released by the reactions. Three hydrocarbon product streams, heavy F-T liquid, medium F-T liquid, and light F-T liquid are sent to the F-T Product Upgrading (F-TPU) Unit while F-T water, a reaction byproduct, is returned to the Gasification Unit and injected into the gasifier or slurried with the petroleum coke. The F-T tail gas and AGR off gas are sent to the gas turbine as fuel to increase electrical power production by 11%.

In the F-TPU Unit, the three F-T liquids are combined and processed as a single feed. In the presence of a hydrotreating catalyst, H_2 reacts slightly exothermally with the feed to produce saturated hydrocarbons, water, and some hydrocracked light ends. The resulting four liquid product streams are naphtha, diesel, low-melt wax, and high-melt wax and leave the EECP facility via tank truck.

The power block consists of a GE PG6101 (6FA) 60 Hz heavy-duty gas turbine generator and is integrated with a two-pressure level heat recovery steam generator (HRSG) and a non-condensing steam turbine generator. The system is designed to supply a portion of the compressed air feed to the ASU, process steam to the refinery, and electrical power for export and use within the EECP facility. The gas turbine has a dual fuel supply system with natural gas as the start-up and backup fuel, and a mixture of syngas from the gasifier, offgas from the AGR Unit, and tail gas from the F-T Synthesis Unit as the primary fuel. Nitrogen gas for injection is supplied by the ASU for NO_x abatement, power augmentation, and the fuel purge system.

The Praxair ASU is designed as a single train elevated pressure unit. Its primary duty is to provide oxygen to the gasifier and Sulfur Recovery Unit (SRU), and all of the EECP's requirements for nitrogen and instrument and compressed air. ASU nitrogen product applications within the EECP include its use as a stripping agent in the AGR Unit, as diluents in the gas turbine where its mass flow helps increase power production and reduce NO_x emissions, and as an inert gas for purging and blanketing. The gas turbine, in return for diluent nitrogen, supplies approximately 25% of the air feed to the ASU, which helps reduce the size of the ASU's air compressor, hence oxygen supply cost.

Acid gases from the AGR, as well as sour water stripper (SWS) off gas from the Gasification Unit, are first routed to knockout drums as they enter the Claus SRU. After entrained liquid is removed in these drums, the acid gas is preheated and fed along with the SWS gas, oxygen, and air to a burner. In the thermal reactor, the H_2S , a portion of which has been combusted to sulfur dioxide (SO_2), starts to recombine with the SO_2 to form elemental sulfur. The reaction mixture then passes through a boiler to remove heat while generating steam. The sulfur-laden gas is sent to the first pass of the primary sulfur condenser in which all sulfur is condensed. The gas is next preheated before entering the first catalytic bed in which more H_2S and SO_2 are converted to sulfur. The sulfur is removed in the second pass of the primary sulfur condenser, and the gas goes through a reheat, catalytic reaction, and condensing stage two more times before leaving the SRU as a tail gas. The molten sulfur from all four condensing stages is sent to the sulfur pit, from which product is transported off site by tank truck.

The tail gas from the SRU is preheated and reacted with hydrogen in a catalytic reactor to convert unreacted SO₂ back to H₂S. The reactor effluent is cooled while generating steam before entering a quench tower for further cooling. A slip stream of the quench tower bottoms is filtered and sent along with the condensate from the SRU knockout drums to the SWS. H₂S is removed from the quenched tail gas in an absorber by lean methyldiethanolamine (MDEA) solvent from the AGR Unit, and the tail gas from the absorber is thermally oxidized and vented to the atmosphere. The rich MDEA solvent returns to the AGR Unit to be regenerated in the stripper.

Environmental RD&T

An important objective of the EECF Project is to explore potential methods to minimize and/or eliminate waste streams to reduce the overall costs and environmental impact of the EECF concept. During Phase I, each of the EECF subsystems were assessed and methods were identified to reduce potential waste streams. The technical risks and barriers around these methods were incorporated into the Phase II RD&T plan to mitigate the identified risks (Phase II RD&T Plan, October 2000).

The leading-edge combination of the Texaco gasification process with modern Fischer-Tropsch or F-T technology and combined cycle power generation allows coals, petroleum coke, and by-product streams to be converted into cleaner transportation fuels and electric power with lower greenhouse emissions.

The Texaco gasification process has been acknowledged by U.S. regulatory agencies and by trade and industry groups as an environmentally attractive technology for processing low-rank carbon or hydrocarbon feedstocks such as petroleum coke. The gasification process converts the hydrocarbon content of these feedstocks into a synthesis gas or syngas. The metals in the low-rank carbon or hydrocarbon feedstock are transformed into an inert slag. Tests were conducted on slag from a petroleum coke-fed Texaco gasifier to verify the expected composition. Additionally, potential markets for the slag were examined in Subtask 2.10.1 (Petroleum Coke Slag Characterization).

Normally, spent F-T iron catalyst would be disposed in landfills. The EECF spent catalyst was examined to determine if it can be fed to the gasifier with the hydrocarbon feedstock (Subtask 2.10.2). In the event that landfilling is required; more information will be needed to determine if the spent catalyst can be landfilled in non-hazardous containment areas. A study (Subtask 2.10.3) was carried out to identify non-hazardous landfill requirements and if the spent F-T iron catalyst will meet government requirements.

The EECF is expected to produce less greenhouse gases than conventional refineries or power generating stations per unit of energy production due to higher thermal efficiency. Total emission of CO₂, sulfur oxides (SO_x), CO, and NO_x are expected to be lower due to higher plant efficiency. To further minimize greenhouse gas emissions, a method to isolate and recover CO₂ from the gas turbine exhaust was investigated by Oak Ridge National Laboratory for the EECF (Subtask 2.10.4).

Petroleum Coke Slag (Subtask 2.10.1)

Petroleum coke slag is a byproduct of the proposed EECF. Traditionally, petroleum coke slag is landfilled. As part of Phase II of the development of the EECF, ChevronTexaco conducted a paper study of potential markets for petroleum coke slag. The petroleum coke slag produced by the proposed EECF may be sold into the construction industry as aggregate, the cement industry as kiln fuel, or the blast grit industry. Any use of the slag is dependent on the final characteristics of the slag and the distance of the proposed EECF to the slag market.

The Texaco Gasification Process

Gasification is the first step in proposed EECF. ChevronTexaco's gasification process has been acknowledged by U.S. regulatory agencies and by trade and industry groups as an environmentally attractive technology for processing low-rank carbon or hydrocarbon feedstocks. For the proposed EECF, petroleum coke will be converted into synthesis gas (syngas), which is largely composed of carbon monoxide and hydrogen. Metals in the petroleum coke leave the gasifier as part of an inert slag. The synthesis gas can be sent to the combined cycle power block to generate electricity and the F-T reactor to produce ultra clean transportation fuels, waxes, and/or lubricants. Additional products from the EECF can include steam and hydrogen. Figure 1, below, shows the Texaco Gasification Process.

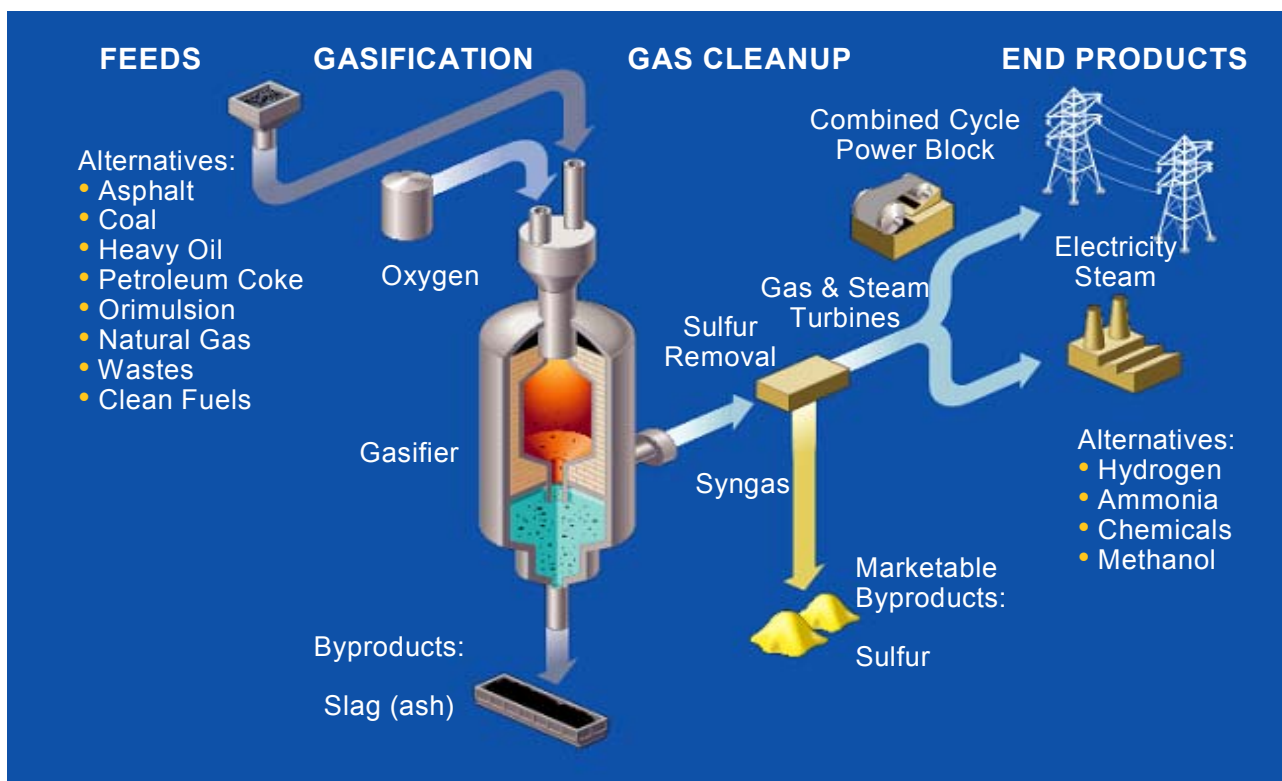


Figure 1. Texaco Gasification Process

Petroleum Coke Slag Characteristics

Petroleum coke slag is a byproduct of the ChevronTexaco Gasification Process. The slag consists of unconverted carbon, trace amounts of hydrogen, and ash containing heavy metals, such as vanadium and nickel. Previous studies have shown the slag is non-hazardous and safe for disposal. Instead of disposing the slag, it may have uses in construction or other commercial products.

While there are slight variations in slag from one gasifier to another based on the petroleum coke feed and gasification conditions, petroleum coke slag tends to have low carbon and hydrogen content. The majority of the slag tends to be ash.

Potential Uses for Petroleum Coke Slag

Petroleum coke slag can be disposed in most landfills. To move towards achieving DOE's goal of a "zero discharge" commercial coproduction plant in the future, ChevronTexaco investigated several potential commercial applications for petroleum coke slag. Some potential uses for petroleum coke slag are listed below:

- * Construction Products (cement aggregate, cinder block, etc.)
- * Fuel (blast smelter feed, cement kiln fuel, etc.)
- * Miscellaneous (blast grit, polishing, etc.)

Depending on the application, the petroleum coke slag may have to undergo preparation before it can be used in any of the following products:

Construction Products

Petroleum coke slag can be used as an aggregate in concrete or other construction products. Petroleum coke slag can also be used in asphalt binder or road bed aggregate. Concrete, for example, consists of a binder material, water, and an aggregate or filler. Concrete can contain up to eighty (80) percent aggregates. Natural aggregates, such as crushed stone and sand, comprise the majority of aggregates used in the United States. The market for construction product aggregates is very large. For example the market for crushed stone averages over one (1) billion metric tons per year. The amount of slag produced by the proposed EECF would amount to approximately 8,000 metric tons per year. The petroleum coke slag can be mixed with other aggregate materials, such as crushed stone, in the final product.

The market for construction products is highly competitive and dependent on the construction industry. High material transportation costs can limit the aggregate to local markets. The price of petroleum coke slag would be discounted to the price of crushed stone. While the price of crushed stone fluctuates based on demand, the constant dollar value has changed little in the last 20 years. In this study, the price of petroleum coke slag is expected to be \$4.00 to 5.50 per metric ton, freight on board (fob) plant. With

shipping and handling charges ranging from \$8.00 per metric ton to over \$15.00 per metric ton, it is extremely important for the market to be located near the proposed EECP. Additionally, consolidation in the cement industry makes it important to partner with a large cement company to ensure market access.

Fuel

Petroleum coke slag contains a small amount of unconverted carbon and sulfur. The slag can be used in blast smelter feed or as cement kiln fuel. Smelting is the process used to make lead. A lead bearing feed is sent to a blast furnace mixed with petroleum coke and/or coal. Cement kilns are used to produce cement. The raw materials, limestone, clay, fly ash, and others, are crushed and fused in the kiln. The product is cooled, gypsum is added, and then it is crushed into fine powder. Like cement smelters, the fuel for cement kilns fuel can be petroleum coke and/or coal.

The relatively low heating value of petroleum coke slag limits the market opportunities. It would be mixed with petroleum coke and/or coal. Most likely, the amount of petroleum coke slag used would be less than 5% of the overall fuel. The price of the slag would be based on its heating value. However, the cost of transporting the petroleum coke slag to a suitable smelter or kiln would eliminate any profits.

Miscellaneous

Other uses for petroleum coke slag include blast grit, polishing, or metal reclamation. Blast grit encompasses a wide variety of media, such as steel shot, aluminum oxide, boron carbide, sand, and corncob grit. The material used is dependent on the needs of the user. An important factor when considering which blast grit material to use includes the number of times the material can be used. For example, while steel shot is more expensive than sand, the steel shot can be used fifty (50) or more times. Sand might last for two (2) to five (5) uses. Petroleum coke slag life cycle would be similar to sand's. Research indicates that petroleum coke slag used as blasting grit could command a price of up to \$20 dollars per metric ton. However, it would be critical to locate a dedicated user and undergo a period of testing.

Petroleum Coke Slag Handling in the EECP

In petroleum coke-based Texaco Gasification plants, petroleum coke slag is sent to landfill from the Slag Handling System. The cost of transportation and the need for dedicated off-takers limits the beneficial use of petroleum coke slag from most Texaco Gasification plants. For the proposed EECP, the Phase I Concept Design has the petroleum coke slag landfilled. Once a suitable EECP site is located, the project team can locate potential users of the petroleum coke slag.

Use of F-T Catalyst in the Gasifier (Subtask 2.10.2)

Safe, cost efficient disposal of the F-T catalyst is important to the proposed EECF. Traditionally, spent iron F-T catalyst is sent to landfills (see Subtask 2.10.3). The F-T catalyst removed from the reactor is in a chemically reduced state and in the absence of its wax coating, would be pyrophoric. To minimize this risk, a small layer of wax is left on the surface of the spent F-T catalyst. The potential presence of aromatics in the wax and/or on the catalyst surface could make the F-T catalyst/wax classified as a hazardous waste. An alternative to landfilling identified during Phase I of the EECF Project is to feed the spent iron-based F-T catalyst to the gasifier. The objective of Phase II was to study the viability of feeding the spent F-T catalyst to the gasifier.

Gasification

As shown in Figure 1, the ChevronTexaco gasification process produces synthesis gas from a variety of hydrocarbon-based feedstocks. In the proposed EECF, petroleum coke will be mixed with water to form slurry. The slurry is mixed with high pressure oxygen and a small quantity of high-pressure steam of which is fed to the gasifier in a specially designed feed injector on the gasifier. The resulting reactions take place very rapidly to produce synthesis gas. Metals in the petroleum coke leave the gasifier as an inert slag.

It is proposed to mix the spent F-T catalyst with the petroleum coke and water slurry. The resulting slurry would then be fed to the gasifier with the oxygen and steam. Wax present on the catalyst surface would be gasified into additional synthesis gas and the remaining iron catalyst would leave the gasifier with the slag. The slag from the ChevronTexaco gasification process can be safely sent to landfills or potentially used in several industrial processes (see Subtask 2.10.1, above).

Spent Fischer-Tropsch Catalyst

The fresh F-T catalyst is a precipitated, unsupported iron catalyst. Upon activation, the catalyst (primarily hematite, Fe_2O_3) forms iron carbides and magnetite (Fe_3O_4). Over time, the catalyst pores may contain waxes, interstitial carbon, iron sulfide, and possibly aromatics. The catalyst is removed from the F-T reactor with a small portion of F-T wax. Solvent extraction can be used to minimize the amount of wax remaining on the catalyst surface prior to disposal. Without the wax coating, the F-T catalyst is pyrophoric.

Feeding the spent F-T catalyst to the gasification section presents two unique challenges. First, the spent F-T catalyst must mix with the petroleum coke and water slurry. Finally, the F-T catalyst must not damage the gasifier injector. Unfortunately, to fully test these two issues requires a larger amount of spent F-T catalyst than is available. Therefore, ChevronTexaco evaluated the feasibility of feeding the F-T catalyst to the gasifier based on past experience and analysis of the spent F-T catalyst.

In the Phase I Concept Report, the proposed EECP was based on feeding 1184 metric tons (1305 short tons per day) of petroleum coke. The amount of spent F-T catalyst that would be sent to the gasification section is very small (less than 0.1%) compared to the amount of petroleum coke. In the proposed EECP, the petroleum coke is ground, mixed with water and pumped as thick slurry to the Gasification Unit. The spent F-T catalyst particles are smaller than the size of the ground petroleum coke and should not affect the life of the gasifier injector. The rheology of the petroleum coke slurry is a key issue (see Task 2.9 Topical Report). The spent F-T catalyst would be mixed with the petroleum coke and water. The small amount of spent F-T catalyst along with F-T water and a proprietary additive should not significantly affect the slurry rheology.

Feeding the spent F-T catalyst to the ChevronTexaco gasifier in the proposed EECP provides another option to landfilling. The small amount of spent catalyst (in relation to the amount of petroleum coke) should not negatively affect the performance of the gasification section.

F-T Catalyst Disposal (Subtask 2.10.3)

The most likely alternative for spent catalyst disposal is landfilling. The Rentech F-T precipitated iron catalyst is an ideal candidate for landfilling due to the absence of hazardous ingredients in the catalyst precursor, primarily hematite (Fe_2O_3). Upon activation, iron carbides and magnetite (Fe_3O_4) are formed. The used catalyst, however, may contain heavy waxes, interstitial carbon, iron sulfide, and possibly aromatics. Since the catalyst is used in a wax slurry medium, one issue is the amount of wax to be disposed of with the catalyst. The catalyst removed from the reactor is in a chemically reduced state and therefore in the absence of its wax coating would be pyrophoric. If solvent extraction were to be used to minimize the amount of wax remaining on the catalyst prior to disposal, the spent catalyst could be classified as hazardous due to the characteristic of ignitability. Therefore, Phase II testing focused on the ability to landfill the spent catalyst with a layer of wax. Due to the potential presence of aromatics in the wax and/or on the catalyst surface, catalyst/wax samples would have to undergo testing to determine whether the material exhibits the toxicity characteristic due to the presence of benzene.

Regulations Governing Solid Waste Disposal in Landfills

The Resource Conservation and Recovery Act (RCRA) of 1976 gave the Environmental Protection Agency (EPA) authority to regulate hazardous waste from generation to disposal. RCRA also set the groundwork for the management of non-hazardous solid waste. In 1984, amendments to RCRA known as the Hazardous and Solid Waste Amendments (HSWA) were implemented. These RCRA amendments required phasing out land disposal of hazardous waste, produced tougher hazardous waste management standards and gave increased enforcement authority to the EPA.

The EPA rules governing solid waste disposal are delineated in the Code of Federal Regulations (CFR), Title 40, Chapter I, Part 261, Subchapter I – Solid Wastes. The applicable regulations for the spent F-T catalyst/wax mixture fall under Subpart C – Characteristics of Hazardous Waste. Of the four characteristics, ignitability, corrosivity, reactivity, and toxicity, the toxicity characteristic is the only one of relevance to the catalyst/wax mixture. Table 1 – Maximum Concentration of Contaminants for the Toxicity Characteristic in CFR, Title 40, Chapter I (not shown) lists the level of contaminants which can render a solid waste hazardous. The contaminants are measured using Test Method 1311 described in “Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,” EPA Publication SW-846. The test is the “Toxic Characteristic Leaching Procedure” (TCLP), EPA 1311/8021B, whereby a sample of the waste is tumbled in a sodium acetate/acetic acid solution for 18 hours and the concentration of the leachate is measured in mg per liter (mg/l) of solvent. The only contaminant in the aforementioned Table 1 which is applicable to the spent F-T catalyst/wax mixture is benzene, which has a limit of 0.5 mg/l.

The State of California requires a more stringent test – the “Waste Extraction Test” (WET). In this test, the sample is tumbled in a 0.2M sodium citrate buffer solution for 48 hours and the concentration of leachate is measured in mg per liter of solvent. The California Code of

Regulations defines the characteristics of toxicity in section 66261.24 of Article 3 under Chapter 11, division 4.5 of Title 22. In subsection(a)(2)(A) of section 66261.24 inorganic materials and their concentration limits are listed in Table II of the California Code of Regulations, Title 22, division 4.5, Chapter 11 (not shown) “List of Inorganic Persistent and Bioaccumulative Toxic Substances and Their Soluble Threshold Limit Concentration (STLC) and Total Threshold Limit Concentration (TTLC) Values”. Copper is included in this list with a STLC of 25 mg/l. The TTLC for copper is 2500 milligrams per kilogram (mg/kg).

TCLP And WET Test Results On a Spent F-T Catalyst/Wax Sample

A sample of reactor wax containing about 11 weight (wt%) of catalyst from Rentech’s bubble column reactor test (RI67) was sent to Del Mar Labs in Irvine, CA to perform TCLP and WET tests on the sample to determine levels of BTEX (benzene, toluene, ethylbenzene, and xylene) and copper. The spent F-T catalyst/wax sample from RI67 was taken from the bubble column reactor at the end of a 10-day test. During this test, approximately 3600 grams of catalyst and wax were removed from the reactor and 3600 grams of catalyst and wax were added back to the reactor to demonstrate the catalyst/wax addition and withdrawal system (EECP Phase II Task 2.1). The activated catalyst added each time was slurried in paraffin wax. With the 7 kilograms (kg) of initial paraffin starting wax, the total amount of paraffin wax added to the system was approximately 10 kg, whereas the total amount of wax removed during the 10-day test was about 34 kg. Therefore the wax contained in the sample sent to Del Mar Labs for analysis was predominantly Fischer-Tropsch wax.

The test results in mg per liter of solvent are summarized in Table 2.10.3-1.

Analyte	TCLP	WET	LIMIT
Benzene	Not Detected	Not Detected	0.5
Toluene	0.0037	0.018	---
Ethylbenzene	Not Detected	Not Detected	---
Total xylenes	Not Detected	0.011	---
Copper	0.64	5.0	25.0 (STLC)

Table 2.10.3-1. Results from TCLP and WET Testing of Spent F-T Catalyst/Wax

Catalyst Disposal Quantities and Cost for A EECP Facility

The spent F-T catalyst/wax would be removed from the reactor through two stages of separation. The primary separator would provide wax containing about approximately 1000 parts per million –weight (ppmw) of catalyst to the secondary separation system. The wax from the secondary separator filtrate would contain approximately 10 ppmw of catalyst. Since the total C₅+ production rate of the proposed EECP is 600 barrels per day (Phase I Preliminary Concept Design Report), the catalyst usage would be about 0.23 pounds per barrel. Therefore, the amount of catalyst removed for landfilling would be about 140 pounds per day. If the concentration of the catalyst in the catalyst/wax mixture sent to the landfill were 30%, the total amount including the wax requiring landfilling would be 465 pounds per day.

The tipping fees for California from the 2000 Solid Waste Tipping Fee Survey ranged from \$21.25 to \$71.00 per ton. For an EECF facility located in California, the costs would range from \$4.94 to \$16.51 per day or from \$0.0082 to \$0.0275 per barrel of C₅+ hydrocarbon production. Transportation costs at \$2.50 per ton-mile at a distance of 50 miles would add \$0.048 per barrel of C₅+ hydrocarbon production. For comparison, the tipping fees for Texas averaged approximately \$24.50 per ton in 2002. For an EECF facility located in Texas, the landfill costs would be approximately \$5.70 per day.

CO₂ Recovery from the Gas Turbine Exhaust (Subtask 2.10.4)

The proposed EECF power generation system based on fueling a gas turbine with synthesis gas produced from petroleum coke, F-T tailgas, N₂, and the CO₂ stripped from the SRU would have a typical wet gas exhaust that contains about nine and one-half percent CO₂. The Oak Ridge National Laboratory (ORNL) has developed a physical adsorbent called the Carbon Fiber Composite Molecular Sieve (CFCMS) [Burchell et al., 1997 & 2000] for separating (removing) CO₂ from a wide variety of gas streams. Because the CFCMS material is electrically conductive it may be regenerated by the passage of an electric current at low voltage [1]. This has been exploited in the patented Electrical Swing Adsorption (ESA) Process [Judkins et al.].

The series of CFCMS billets were manufactured from 400 grams of 400 micrometer length isotropic pitch fiber (Anshan East Asian Carbon Company, China), and 120 grams of powdered phenolic resin type Durez 7716 (Occidental Chemical Co.). The mix was slurry molded and the resultant green forms dried for 24 hours at 333K (140°F) in air, cured for 24 hours at 393K (248°F) in air, and carbonized at 923K (1202°F) in flowing N₂ for 4 hours. The parts were thermally activated at 1113K (1580°F) in flowing CO₂ for various lengths of time as reported in Table 1.1.

Table 2.10.4-1 Processing details for CFCMS billets O-1 through -4.

Billet Identity	Activation time (hours)	Burn-off (%)
SMM-26	48	30.1
O-1	28	24.8
O-2	36	37.8
O-3	120	56.4
O-4	75	42.9

The burn-off reported in Table 1.1 is the mass loss due to gasification of the carbon structure during activation represented as a fraction of the original billet mass. Billet O-3 was judged to have excessive burn-off and was not sufficiently durable to be assembled to an ESA cell. Consequently it was discarded. Three of these billets were assembled into ESA cells.

The process of thermal activation develops the micropore (pore size <2 nanometers) network and results in a marked increase in the micropore volume and surface area. Micropore volume and surface area were determined for the billets from N₂ adsorption isotherms (measured at 77K/-321°F) using the BET method for surface area and the Dubinin-Redushkavich method for micropore volume and size.

Objective

The purpose of this work was to modify and/or develop a version of CFCMS capable of removing CO₂ from a typical dry turbine exhaust gas shown in Table 2.10.4-1, and to apply the ESA process and CFCMS material to the exhaust or gas stream. Because this investigation is at ambient conditions and the moisture in the gas turbine exhaust would probably condense within the set-up, it was decided to use the dry exhaust gas composition.

Table 2.10.4-2: Dry Exhaust Gas Composition and Derived Density

Gas	% Mol	% Mass	Density (g/l)	Density frac
CO ₂	10.05	14.65	1.7973	0.2633
O ₂	10.11	10.75	1.3007	0.1398
N ₂	79.09	73.60	1.1233	0.8267
Ar	0.75	1.00	1.6240	0.0162
Total	100.00	100.00		1.2461

Project

The project was divided into four separate activities: (1) CFCMS Optimization; (2) CO₂ Capacity Determination; (3) Design Data Acquisition, and (4) the Design of a CO₂ Removal Device.

CFCMS Optimization

Several CFCMS billets were fabricated under varying conditions and their micropore characterizations were determined. Typical physical characteristic data are shown in Table 2.10.4-2. These CFCMS billets were machined and assembled into ESA cells for testing, which included dynamic gas separation testing, pressure drop determination, and regeneration studies.

Table 2.10.4-3: Billet Processing details and their Micropore Characterization Data

Billet Identity	Activation time (hours)	Burn-off (%)	BET Surface Area, m ² /g	D-R Micropore Volume, cm ³ /g	D-R micropore size, nm
SMM-26	48	30.1	1659	0.61	2.2
O-1	28	24.8	1326	0.49	2
O-2	36	37.8	1790	0.63	2.4
*O-3	120	56.4	-	-	-
O-4	75	42.9	1757	0.61	2.4

*O-3 was not characterized because it was judged to have excessive burn-off.

CO₂ Capacity Determination

Single gas isotherms were measured gravimetrically using a Hiden IGA gravimetric analyzer for CO₂, N₂ and O₂ over the pressure range 0-1300 millibar (mb) and at nominal temperatures of 298.15K (25°C), 323.15K (50°C), 348.15K (75°C), and 373.15K (100°C). Over these ranges, the amount of gas adsorbed increased with increasing pressure, but decreased with increasing temperature. Thus, at 1300 mb, the amount of CO₂ adsorbed decreases from >10 wt% to <3.5 wt% over the temperature range 297.15K (24°C) to 366.15K (93°C). In comparison with CO₂ adsorption behavior, significantly

less (~ 10% of CO₂) N₂ is adsorbed for a given temperature and pressure indicating that CO₂ should be preferentially adsorbed in the presence of N₂.

Design Data Acquisition

Breakthrough curves were developed in cylindrical ESA cells (10.8 centimeters [4.25 inches] diameter and lengths varying from 17.8 to 22.9 centimeters [7 to 9 inches]). All experiments were performed at ambient conditions (1 atmosphere and room temperature). The CO₂-dynamic capacity of the CFCMS material and the pressure drop were measured. In addition several regeneration tests were made to simulate the ESA process.

Table 2.10.4-3 shows the dynamic capacity, in grams (g), of CFCMS at varying activation levels and flow rates, in liters per minute (l/m). Although the variation in the capacities appears small, the optimum capacity was shown to be at a flow rate of ~ 10 liters per minute and an activation level at 30 +/- 5% burn-off.

Table 2.10.4-4: Dynamic Capacity (g-CO₂/g-CFCMS) Vs Flow Rate

Flow rate, l/m	Burn-off			
	24.8%	30.1%	37.8%	42.9%
1.0	0.0136	0.0128	0.0164	0.0097
3.0		0.0159		
5.0	0.0176	0.0165	0.0179	0.0131
7.0		0.0200		
10.0	0.0170	0.0177	0.0179	0.0119
15.0	0.0184		0.0161	0.0119
15.5		0.0193		
19.5	0.0171			
20.0			0.0143	

The pressure drop across the cell was very low. At all four activation levels and for flow rates 1 – 20 l/m, the pressure drop range is 0.003 – 0.02 pounds per square inch per inch (psi/inch).

The viability of the ESA process depends on a well-defined and optimized regeneration step. Consequently, several regeneration schemes were investigated. The efficiency of a regeneration step was determined by how much energy and regenerant gas (N₂) was required to complete the regeneration and prepare the cell for the next adsorption (production) cycle.

1. Power only: Feed was stopped at breakthrough. Power (low voltage - 6 volts, ~12 amps) was passed through cell for a given time (5 or 6 minutes). Then N₂ flowed through until regeneration was completed. Large amount of energy and N₂ were consumed to complete regeneration with low CO₂ concentration in the effluent.

2. Temperature only: Feed was stopped. Power was passed through the cell until its temperature was $\sim 343.15\text{K}$ (70°C), and this was maintained by switching the power on and off until regeneration was complete with N_2 flow. Excessive energy and large amount of N_2 were used with low CO_2 concentration in the effluent.
3. Vacuum only: Feed was stopped. Full vacuum (~ 30 inches mercury [in Hg]) was applied to the cell for 3 or 5 minutes, followed by N_2 flow to complete regeneration. Tests were also carried out under partial (~ 10 or 20 in Hg) vacuum. Higher CO_2 concentration was in effluent but large amount of N_2 was needed to complete the regeneration.
4. Temperature and Vacuum: Feed was stopped. Power was passed through the cell to raise and maintain the temperature to $\sim 343.15\text{K}$ (70°C) and maintain it there. At 343.15K (70°C), full vacuum was applied to the cell for 2 minutes. When the vacuum was stopped, N_2 flow was initiated to complete the regeneration. The temperature was kept at $\sim 343.15\text{K}$ (70°C) until the cell was fully regenerated. The experiment was repeated with partial vacuum. Full vacuum produced the highest relative concentration of CO_2 in the effluent with high-energy consumption but low N_2 was used.
5. Power and Vacuum: Feed was stopped. Power and vacuum were simultaneously employed for a given time. The test was repeated under partial vacuum. The Power and Vacuum condition produced CO_2 in effluent comparable to (4), with lower energy consumed and low N_2 used.

The results indicated that Power and Vacuum regeneration was the most attractive regeneration process.

In all the regeneration steps, there was negligible effluent flow without the flow of a regenerant (purge) gas, N_2 . Also because of the experimental set-up, the effluent gas could not be analyzed when the cell was under vacuum.

A series of confirmation tests were also conducted. CFCMS activated at 31.2% burn-off (within the optimum $30 \pm 5\%$) and optimum feed flow rate of 10 l/m were used in confirmation tests. Power-and-Vacuum with partial (ballast system at 18 in Hg) vacuum was the regeneration step. Table 2.10.4-4 shows the result of the tests. In the first two tests, power (low voltage) and vacuum were used for the first 5 minutes, followed by N_2 flow at 2.0 l/m or 1.0 l/m to complete regeneration. In the third, after the power and vacuum were on for 5 minutes, the temperature rose to and was maintained at 343.15K (70°C) as N_2 at 1.0 l/m was used to complete the regeneration. In the last two tests, no vacuum was used. Power and N_2 employed on until the temperature was 343.15K (70°C), then the power was eliminated and N_2 was continued to complete the regeneration.

Table 2.10.4-5: CFCMS Capacity and Regeneration @ 31.2% Burn-off and feed @ 10 l/m

Test Identification	g CO ₂ /g CFCMS	g CO ₂ /l CFCMS	l-scrubbed/l-N ₂
Pow/Vac - 5 min, N ₂ @ 2.0 l/m	0.0266	4.31	0.891
Pow/Vac - 5 min, N ₂ @ 1.0 l/m	0.0266	4.31	0.836
Pow/Vac - 5 min, T-70, N ₂ @ 1.0 l/m	0.0254	4.12	0.880
Pow T-70, N ₂ @ 2.0 l/m	0.0272	4.40	0.839
Pow T-70, N ₂ @ 1.0 l/m	0.0266	4.31	0.748

Since the feed rate was the same, the capacities were 0.0265 (+/- 3%) g CO₂/g CFCMS, which are much higher than the values in Table 2.10.4-3. The efficiency of regeneration (liters of feed processed/liters of N₂ to complete the regeneration) did not increase significantly with the addition of either vacuum only or vacuum with constant temperature at 343.15K (70°C) compared to just heating the cell to 343.15K (70°C). However, the CO₂ concentration in the effluent from the three tests with vacuum was about 20 wt% compared to 15 wt% for the final two.

Design of a CO₂ Removal Device

The design of the CO₂ removal device for a gas turbine exhaust stream was based on the removal of 1.0 kg of CO₂ per minute by adsorption in a CFCMS column. This is equivalent to processing dry feed gas at 6.626 kg or 5.478 m³ per minute. Assuming that the adsorption was at optimum conditions (CFCMS @ 31.2 % burn-off and feed rate @ 10 l/m), the required amount of CFCMS material is 1.068 cubic meters (m³). To ensure continuous CO₂ removal, a four-column system has been selected. During the operation, one column is under adsorption, the second is under regeneration, the third is cooling down to ambient conditions, and the fourth is on stand by.

Because the vacuum system was complex and showed no marked advantage, the regeneration system selected was the temperature only, in which power is passed through the column to increase and maintain its temperature at 343.15K (70°C). The regenerant (purge) gas is N₂, the worst-case scenario. The regeneration cycle was not optimized. However, by combining countercurrent flow and operating the column at 343.15K (70°C), it was assumed that N₂ flow at 20 l/m would regenerate the column in the time it takes to saturate it with CO₂. Based on these assumptions, the N₂ required is 10.956 m³ per minute, the energy input to increase the column temperature to 343.15K (70°C) is 11.544 megajoules (MJ), and the power to maintain the temperature at 70°C is 8.215 kilowatts (kW).

The raffinate (CO₂-free effluent from adsorption column) may be used in the initial regeneration. This scenerio reduces the N₂ required to 5.898 m³ per minute. There may also be heat exchanged by passing N₂ through the cooling column and into the heated column during regeneration. The resultant energy saving, however, was not computed.

Another approach to the design is to assume equilibrium adsorption at $\sim 25^{\circ}\text{C}$ and one atmosphere and equilibrium desorption at high vacuum and elevated temperature. This represents the best-case scenario. The equilibrium desorption values at 600 mb and 70°C were used in this case. Under these conditions, only 25 wt% of the CO_2 adsorbed (0.25 kg/minute) is left in the CFCMS column after the vacuum and temperature regeneration. At the same flow rate assumed above, only 25 wt% of the purge gas ($2.739 \text{ m}^3/\text{minute}$) is needed. This is less than the CO_2 -free gas stream produced during the adsorption of CO_2 , which can be used to regenerate the column. However, some pure N_2 may be needed to cool the column to ambient and remove the O_2 and Ar that are adsorbed from the CO_2 -free stream.

The energy input to increase and maintain the column temperature at 70°C and maintain it there remains the same, 11.544 MJ and 8.215 kW, respectively. Recycling will reduce the energy load.

CFCMS Observations

1. The CFCMS cell removes all the CO_2 from the feed until there is a breakthrough.
2. The highest dynamic capacity of the CFCMS is at an activation level of 30 +/-5% burn-off.
3. The highest dynamic capacity was obtained with a feed gas flow rate of 10 liters per minute.
4. Confirmation tests at 31.2% burn-off and 10 l/m yielded the highest dynamic capacity of 0.0265 g- CO_2 /g-CFCMS (4.31 g- CO_2 /l-CFCMS).
5. The dynamic capacity of the CFCMS material is high. At feed flow rate of 5 l/m, it is 85% of the equilibrium value and 60% at 11.5 l/m.
6. The pressure drop across the CFCMS cell was very low. At four activation levels (24.8, 30.1, 37.8, and 42.9% burn-off) and the flow range of 1 – 20 l/m, the pressure drop varied from 0.003 to 0.02 psi/inch of cell.
7. CFCMS cell saturated with CO_2 cannot be completely regenerated without the flow of a purge gas.
8. Under ideal conditions and the most favorable equilibrium regeneration (full vacuum and high temperature), the effluent gas contains $\sim 68 \text{ wt}\%$ CO_2 . If this gas stream is processed in a second adsorption-desorption cycle the effluent contains $>95 \text{ wt}\%$ CO_2 . However, during the various regeneration processes investigated in this work, the highest initial effluent CO_2 concentration was $\sim 20 \text{ wt}\%$.
9. Power (low voltage) into the CFCMS cell improves the regeneration efficiency, but power input alone cannot completely regenerate the CFCMS cell without a purge gas.
10. Power and vacuum enhance the regeneration efficiency; however, the process is complex and difficult to demonstrate within our experimental set-up.
11. An adsorber to remove 1 kg of CO_2 per minute requires a 1.068 m^3 CFCMS column
12. To ensure continuous removal of CO_2 , a four-column system is recommended.
13. Without recycle in the worst-case scenario, regenerating the CFCMS requires 10.956 m^3 per minute of N_2 , 11.544 MJ to increase the column temperature to 70°C , and 8.215 kW to maintain the temperature at 70°C .

14. With recycle, only 5.898 m³ per minute of N₂ is required. The energy consumption is also reduced.
15. In the best-case scenario, only a relatively small amount of pure N₂ is required for cooling and for the final column regeneration. The energy consumption to increase and maintain the column temperature becomes the main cost in the regeneration.

Conclusions

Task 2.10 of the EECF Project focused on minimizing the environmental impact of the potential waste streams from the proposed EECF. Novel alternative uses for the gasifier slag, spent F-T catalyst, and CO₂ from the gas turbine exhaust were examined.

Petroleum coke slag has many alternative uses besides landfilling. The heating value of the unconverted carbon in the slag allows it to be used in cement kilns and blast smelters. The petroleum coke slag can also be used in a variety of construction industry products including concrete and roads. The most interesting use may be as blast grit, where the slag may command prices of up to \$20 per metric ton. However, all of these uses are dependent on the project location and the final petroleum coke slag characteristics. It is critical to find an alternative use that is relatively close to the proposed gasifier. Otherwise, shipping costs could eliminate any profit from the sale of the slag.

Spent F-T catalyst can be sent to the gasifier as part of the petroleum coke slurry or sent to landfill. Further testing with a larger amount of spent F-T catalyst then is currently available may be required prior to using the spent F-T catalyst in the petroleum coke slurry. Under current EPA and the more stringent California rules governing landfill disposal, the Rentech spent F-T catalyst/wax mixture qualifies for classification as a non-hazardous waste. The cost of transporting and landfilling the spent F-T catalyst/wax mixture should cost less than \$0.10 per barrel. In the event that landfilling hydrocarbons is precluded in the future, the catalyst/wax mixture would require incineration followed by landfilling the ash. This approach would increase the cost significantly.

Initial testing conducted at ORNL has demonstrated the potential of a CFCMS to meet the objectives for the removal of CO₂ from the exhaust of a gas turbine. Additional development and configuration design is required to optimize the CFCMS system before it can be incorporated into an EECF or Commercial Coproduction Plant (CCP).

Based on the results in Phase II Task 2.10, the only change to the Phase I Conceptual Design Basis would be feeding the F-T water to the gasification slurry instead of directly pumping the F-T water to the gasifier. This would result in a lower cost to the gasification section since a high pressure pump would no longer be needed.

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List of Acronyms and Abbreviations

AFDU	Alternative Fuels Development Unit	m ³	cubic meter
AGR	Acid Gas Removal	mb	millibar
Ar	Argon	MJ	megajoule
ASU	Air Separation Unit	MDEA	Methyldiethanolamine
BTEX	benzene, toluene, ethylbenzene, and xylene	mg/kg	milligrams per kilogram
CCP	Commercial Coproduction Plant	mg/l	mg per liter
CFCMS	Carbon Fiber Composite Molecular Sieve	N ₂	Nitrogen
cm	centimeter	nm	nanometer
CO	carbon monoxide	NO _x	nitrogen oxides
CO ₂	carbon dioxide	ppmw	parts per million–weight
DOE	U.S. Department of Energy	psi	pounds per square inch
EPA	U.S. Environmental Protection Agency	RCRA	Resource Conservation and Recovery Act of 1976
ESA	Electrical Swing Adsorption	RD&T	Research, Development, and Testing
F-T	Fischer-Tropsch	SO ₂	sulfur dioxide
F-TPU	Fischer-Tropsch Product Upgrading	SO _x	sulfur oxides
Fe ₂ O ₃	hematite	SRU	sulfur recovery unit
Fe ₃ O ₄	magnetite	STLC	Soluble Threshold Limit Concentration
g	grams	SWS	sour water stripper
GE	General Electric	TCLP	Toxic Characteristic Leaching Procedure
H ₂	hydrogen	TES	Texaco Energy Systems LLC
H ₂ O	water	TTLC	Total Threshold Limit Concentration
H ₂ S	hydrogen sulfide	WET	Waste Extraction Test
HRSG	heat recovery steam generator	wt%	weight percent
HSWA	Hazardous and Solid Waste Amendments to the RCRA		
IGFT	ChevronTexaco Integrated Gasification Fischer-Tropsch		
in HG	inches mercury		
K	Kelvin		
Kg	kilograms		
kW	kilowatt		
KBR	Kellogg Brown & Root		
l	liter		
l/m	liters per minute		
m	meter		